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LETTER DATED 11 JUNE 1992 FROM THE CHARGE D'AFFAIRES A.I. OF NORWAY ADDRESSED TO THE PRESIDENT OF THE CONFERENCE ON DISARMAMENT, TRANSMITTING A RESEARCH REPORT ENTITLED "VERIFICATION OF A CHEMICAL WEAPONS CONVENTION: RECOMMENDED OPERATING PROCEDURES FOR SAMPLING AND SAMPLE HANDLING, PART XI"

I have the honour to transmit to you a research report entitled "Verification of a Chemical Weapons Convention: Recommended Operating Procedures for Sampling and Sample Handling, Part XI". This research report represents a further contribution of Norway to the negotiations in the Conference on Disarmament on a Chemical Weapons Convention.

I would appreciate it if the report could be circulated as an official document of the Conference on Disarmament as well as the Ad Hoc Committee on Chemical Weapons.

(Signed) Bjørn Skogmo
Ambassador
Chargé d'affaires a.i.

RESEARCH REPORT ON VERIFICATION OF A CHEMICAL WEAPONS CONVENTION

RECOMMENDED OPERATING PROCEDURES FOR SAMPLING AND SAMPLE HANDLING

PART XI

ROYAL NORWEGIAN MINISTRY OF FOREIGN AFFAIRS
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SUMMARY

The collection and handling of samples is an important part in verification of a chemical weapons convention. Reliable results from sophisticated analytical equipment can only be obtained if the samples have been collected and treated properly. This report gives complete procedures for sampling and sample handling in connection with both verification of alleged use and verification of alleged production in a chemical facility.

The recommended operating procedures presented here are based on extensive field testing at the Norwegian Defence Research Establishment during the last ten years in order to reveal problems which do not arise in laboratory experiments.

The procedures deal with all parts of sampling and sample handling, including localization of the contaminated area, the amounts and sizes of samples which should be collected, and the preferred sample materials. Methods for packing, securing and transport of samples are also described together with sample handling in the laboratory.

One of the most important parts of the sampling procedure is proper documentation of the samples, sampling site and sampling procedures. A form has therefore been drawn up in which all information obtained during sampling should be written. A transport log which should follow the samples from the sampling site to their destination has also been worked out and is presented in this report.

The last part of sample handling is treatment of the sample in the laboratory. Procedures for sample homogenization, splitting and preparation before the final analysis have been developed.

It is important to have proper equipment available for sample collection and sample handling. . A list of suitable equipment has therefore been included in this report.

1 INTRODUCTION

In order to implement a chemical weapons convention successfully, it must be possible to verify violation of any part of the convention. Procedures should be available in connection with verification of alleged use of chemical weapons, existing stocks, their destruction, and the non-production of chemical weapons by the chemical industry. Verification of alleged use requires somewhat different procedures from verification of a production facility. This paper presents one complete procedure for each of these separate activities, even if parts of the procedures are similar.

A complete verification procedure may be divided into several parts, including methods for localization of the contaminated area, sampling, sample handling including preservation, packaging, coding and documentation, transport, sample handling in the laboratory and laboratory analysis. In the context, the laboratory could either be a designated laboratory or the laboratory of the Technical Secretariat. All these stages must be carried out properly in order to ensure that verification is as reliable as possible.

The recommended operating procedures for sampling and sample handling, which are crucial steps in any verification procedure, are based on several years of extensive study. The reliability of verification is influenced by various factors such as where the samples are collected, the kinds of sample materials chosen and how the samples are treated before reaching the laboratory. During transport, it is important to ensure that the transport log is properly completed in order to maintain un unbroken chain of custody.

All the procedures recommended have been thoroughly tested in field trails, which are used to test all methods and techniques and make them functional for field use. Efforts have been made to develop procedures using readily available, unsophisticated equipment.

2 ALLEGED USE

2.1 Verification of location

Upon arrival in an area where use of chemical warfare agents is alleged to have taken place, the position (coordinates) should be checked against the information given by the requesting state party to ensure that the correct area is examined. This should be done by comparing the terrain with a map, or by using navigational aids such as the global positioning system (GPS).

2.2 Localization of the contaminated area

When the reported position has been verified according to the methods in Chapter 2.1, the area possibly contaminated with chemical warfare agents should be localized. This localization should be based on information collected in the following ways:

- Possible eye witnesses should be interviewed to obtain information about the attack, the existence of bomb craters, remnants from shells etc or about animals or people affected by the attack.

- Signs of battle activity, remnants of bombs, shells, etc., and the position of injured animals or people should be inspected and taken as an indication of the position of the contaminated area.

- A limited area may be examined using hand held detection devices such as the chemical agent monitor (CAM) and detection paper or reconnaissance vehicles (e.g. Fuchs). Such devices should also be used to define the pattern of contamination in the area. To make it easier to define the upwind edge of the contaminated area, the examination should

be started upwind of the area and be continued in the wind direction to the other side.

2.3 Sampling

2.3.1 Recommended sampling sites

When the contaminated area has been localized and examined in accordance with the methods described in Chapter 2.2, the sampling sites should be selected using the following criteria:

- Samples should be collected within the area where the highest concentrations of chemical agents are believed to be found, i.e. in the area where the highest concentrations have been indicated by the localization procedures (Chapter 2.2).

- Weather conditions influence the persistence of chemical warfare agents. Samples should therefore be collected from sites where weather conditions have least influence on the recovery of the various agents.

- If it is impossible to define the contaminated area, samples should be collected randomly over the whole area where the use of chemical warfare agents was alleged to have taken place.

2.3.2 Recommended sample materials

When samples are collected for verification of the use of chemical warfare agents, it is important to consider the sample materials which should be selected:

- Sample materials which adsorb the chemical warfare agents efficiently should be selected.

- It must be possible to desorb the agents from the sample materials for analysis.

- The sample material should not have properties which accelerate the breakdown rate of the chemical warfare agents.

Several different types of sample materials may be found in a battlefield environment, and the recommended types are listed below:

- *Liquids from bombs or shells*. If bombs or shells containing liquids are found, they should be sampled because the liquid is likely to be a chemical warfare agent.

- *Other liquids*. Liquids or damp spots found in the contaminated area should be checked with detection paper for the presence of chemical warfare agents. If the test result is positive, samples should be collected for further analysis.

- *Filter canisters*. Filter canisters used by personnel exposed to, or believed to be exposed to, the chemical warfare agents should be collected.

- *Textile materials, leather*. Samples of textile materials or leather used by personnel exposed to the chemical attack should be collected because these materials have been shown to adsorb chemical warfare agents efficiently.

- *Polymers*. Polymers such as rubber, plastic, paint etc. also adsorb chemical warfare agents efficiently and should be collected if they are found in the contaminated area.

- *Environmental samples*. Environmental samples such as snow, sand, soil, vegetation, surface water, concrete, etc. should be collected from several sites in the contaminated area. Since experiments have shown low vertical diffusion rates for chemical warfare agents in solid samples, only the top layer (about 2 cm) should be collected.

Biological samples may be of interest, but may be more difficult to collect for religious or ethical reasons. Such sampling also requires special techniques and should be carried out by medical personnel.

- *Body liquids*. Blood, urine or other body liquids should be collected from humans or animals exposed to the chemical warfare agents, since hydrophilic compounds will be concentrated in such liquids. The cholinesterase activity in blood should be measured to indicate possible exposure to nerve agents.

- *Cadavers*. Tissues or organs from dead humans or animals believed to have been exposed to the chemical warfare agents should be collected. The organs which provide most information are the kidneys, liver, heart and fatty tissue. These organs concentrate lipophilic compounds. Absorbed chemical agent my also be found in hair samples. In addition, samples from the skin or lungs should be collected to document any damage caused by vesicants. Nervous tissue could be collected to document exposure to nerve agents or other agents affecting the nervous system.

2.3.3 Numbers and sizes of samples

It is important to consider the number of samples which are required to allow correct conclusions to be drawn from the results of the sample analysis. We normally have little or no knowledge of the background concentrations in environmental and biological samples. Controls are therefore of the utmost importance if we are to obtain conclusive results from the analyses of such samples.

The following instructions should be followed in collecting environmental and biological samples:

- At least two liquid samples from bombs or shells should be collected in separate containers.

- In order to achieve at least 90 % probability of verifying a chemical attack, 20 environmental or battlefield samples should be randomly collected per area of 100 000 m^2 where the use of chemical weapons is alleged to have taken place. Fewer samples are necessary if clear evidence of such an attack has been obtained using devices such as CAM or detection paper. Three controls should be collected well outside the contaminated area and treated in the same way as the samples. The controls should be of a matrix as similar as possible to that of the samples.

- If it is possible to collect biological samples from humans or animals, at least two samples should be taken from each individual. If only one individual is available, more (10) samples should be taken. Body liquids from individuals not exposed to chemical weapons should also be collected as controls.

- The sample size should be about 50 g in the case of environmental samples (snow, water, sand, soil, concrete, etc) and about 20 cm^2 in the case of polymers, clothing or leather. About 10-50 g of body liquids or organs from dead humans or animals is considered sufficient. If the

samples need to be split before analysis, larger samples should be collected to get the above-mentioned sizes of each sub-sample.

2.3.4 Sample containers

The most satisfactory sample containers are glass bottles with leakproof caps, but mylar bags or metallized plastic bags may also be used. The containers should fulfil the following requirements:

- The containers should not release any chemicals which could contaminate the samples.

- It should not be possible for volatile compounds to escape from the containers.

- The containers should not consist of materials which strongly adsorb chemical warfare agents

- The containers should not accelerate the breakdown rate of chemical warfare agents.

- Gas samples should be passed through a column containing an adsorbent (e.g. Tenax) and should be placed in gas-tight containers.

2.3.5 Sampling procedure

To ensure the integrity of the samples, they should be collected by the inspection team itself. A spoon, spatula, scissors, knife, scalpel, scoop and pipette should be available for sample collection. In addition, personnel carrying out the sampling should wear full protective equipment. The sample containers and sampling equipment should be decontaminated after sampling by washing with 5 % sodium

hydroxide in a mixture of 2-propanol and water (1:1). Fullers' earth may also be used.

2.4 Field analysis

Field analysis can give the first indication of which chemical warfare agents are present in the area. This information may make it easier to select the best method for sample preparation and analysis, and thereby give the most reliable results.

The following techniques may be used for field analyses:

- Thin layer chromatography (TLC).

  Samples collected as described above are extracted with a small volume of dichloromethane, 20 μl of which are applied to silica TLC plates. Use the following mobile phases:
  cyclohexane:ethylacetate:acetone = 5:3:3 for sarin, soman and tabun,
  methanol:acetone = 4:1 for VX,
  n-hexane:methanol:dichloromethane = 7:1:2 for lewisite, clark and adamsite, and toluene for mustard gas.

  The nerve agents sarin, soman, tabun and VX are detected by an enzymatic reaction. Spray with cholinesterase (250 IU in 100 ml phosphate buffer pH=7.4), warm gently (30°C-40°C for 5 min) and spray with a mixture of 1-naphtylacetate (250 mg) and fast blue salt (400 mg) in ethanol (100 ml). White spots on a red background indicate the presence of nerve agents. Note that this reaction is very sensitive. If the concentrations of nerve agents in the samples are too high the spots become large and difficult to define.

Lewisite, clark and adamsite are detected by oxidation with potassium permanganate. Spray with potassium permanganate (40 mg in 100 ml water). White to yellow spots indicate the presence of these compounds.

Mustard gas is detected by spraying with 4(4'-nitrobenzyl)-pyridine (5g in 100 ml ethanol), heating to high temperature (150°C) and spraying with sodium hydroxide (4 g in 100 ml water:methanol=1:1). Blue spots which disappear quite rapidly indicate the presence of mustard gas.

The retention factors using the mobil phases described above are: tabun: 0.43, sarin: 0.34, soman: 0.48, VX: 0.62, lewisite I: 0.27, clark I: 0.52, clark II: 0.51, adamsite: 0.15 and mustard gas: 0.71.

- Information obtained by CAM should be used as an indication of the presence of nerve agents or mustard gas.

- Detection paper should be used on droplets or damp spots to give a indication of the presence of nerve agents or mustard gas.

- Other instruments should be considered as soon as miniaturization has made them available as mobile field equipment.

2.5 Sample handling

2.5.1 Sample sealing

The samples should be sealed and secured immediately after collection to prevent loss or tampering before analysis. The plastic bags and glass bottles used for sampling should be sealed with a lead seal to prevent tampering during transport and storage. It is also important that the containers used for sampling are airtight to prevent any loss of sample.

2.5.2 Sample coding and documentation

Each sample should receive a unique identification code, and all information should be recorded in a sample documentation form with corresponding coding. A three-page form has been drawn up for use by the sampling team and is shown as annex 1 to this report. The following information should be recorded in the form:

- On the first page, information on the sampling procedure is entered, including the reason for sampling, an indication of the priority or importance of the samples, description of samples and sampling site, sampling methods and results of field analyses.

- On the second page, a sketch of the sampling site should be drawn, indicating topography, any bomb craters observed and wind direction. The positions of the sampling sites should also be indicated on this sketch.

- The third page should be filled in if information on the attack is available. Reports from eyewitnesses, casualties, effects on vegetation and meteorological data such as temperature, wind and precipitation since the attack should be entered here. If there are casualties, reports from medical inspections should also be entered.

Two copies of the form should be made, one of which should accompany the samples to the Technical Secretariat, while the other should be kept by the sampling team.

2.5.3 Sample preservation

All samples should be treated as soon as possible after collection, to ensure that the chemical warfare agents are still present on arrival at the laboratory. The appropriate treatment varies according to the origin of the samples. To ensure the integrity of the samples, in some situations, no treatment is desirable. Sample preservation

should be performed outside the contaminated area in order to prevent cross-contamination of the samples and contamination of the equipment.

- As an easy means of field preservation, aqueous samples may be passed through a cartridge filled with a polymer C_{18} adsorbent (Analytichem International Inc.) which retains chemical warfare agents. The cartridge may then be transported to the laboratory for subsequent elution and analysis. Solid samples may be extracted with water and the extract passed through the C_{18} cartridge in the same way.

  Procedure:

  Wash the solid sample with 50 ml water and pass the sample through a 200 mg C_{18} cartridge which has been pre-wetted with 0.5 ml methanol and 5 ml water. In order to prevent clogging of the cartridge, samples containing large amounts of particulate matter should be filtered through a 20 μm pore-size frit filter or through a Whatman microfibre filter grade GF/A.

- Phosphonic acids, which are decomposition products from nerve agents, are preserved by sorption onto an aminopropyl weak anion exchanger (NH_2). Cartridges filled with 100 mg NH_2 material (Analytichem International Inc.) are attached after the C_{18} cartridges and the sample solution is passed through the combined cartridges. The chemical warfare agents are retained on the C_{18} cartridge (top) and the phosphonic acids on the NH_2 cartridge (bottom).

- Organs or tissues from humans or animals should be placed in ethanol for preservation during transport to the laboratory.

2.6 Sample transport

All samples should be properly secured to avoid injury to personnel handling the samples. This means that all samples should be transported surrounded by activated charcoal in a solid shock-resistant container. In addition, the samples should be treated in accordance with the following guidelines:

- To prevent degradation of chemical warfare agents during transport and storage, both the untreated samples and the C_{18} cartridges should be kept cold, preferably in a box filled with dry ice (-78.5°C). A freezing mixture, for example sodium chloride:ice = 1:3 (-21.3°C) or calcium chloride:ice (min. -55°C) could also be used.

- Liquid samples should not be frozen but should be transported cold in an insulated box with cooling elements.

- Each container should be properly packed and labelled according to the "Technical Instructions for the Safe Transport of Dangerous Goods by Air" (ICAO Doc 9284-AN/905). The shipping names: poisonous solid n.o.s (not otherwise specified) or poisonous liquid n.o.s in class 6.1 (UN no. 2811 and UN no. 2810, respectively) may be used for environmental samples containing only traces of chemical warfare agents. These compounds may be transported by passenger aircraft, except for those with an inhalation toxicity of Packing Group I (Great danger). It should be noted, however, that only boxes which are type approved for transport of dangerous goods may be used. A completed copy of the Shipper's Declaration for Dangerous Goods is shown in annex 2 to this report.

- A transport log should follow each parcel and be filled in by personnel in charge of each stage of the transport from the sampling site to the destination in order to maintain an unbroken chain of custody. The maximum permissible temperature during transport should be filled in by

the sampling team as information for personnel in charge of transport. A transport log has been drawn up and is shown as annex 3 to this report.

2.7 Sample handling in the laboratory

When the samples arrive at the laboratory, they should be treated in such a way that all information contained in the samples remains intact until the final analysis. This means that the following instructions should be followed:

- Upon arrival at the laboratory, the samples should be stored in a safe and tamper-proof place. Solid samples should be stored in a freezer at -20°C or lower and liquid samples preferably at +4°C in a refrigerator. Biological samples (including body liquids) should also be stored in a freezer, but serum or plasma should be separated from the blood samples before storage. The analyses should be carried out as soon as possible. If re-coding is necessary, all information given on the sample documentation form and in the transport log should be linked with the new coding.

- Before the main sample is split into sub-samples, it should be properly homogenized. Dry particulate samples such as sand or dry soil should be homogenized by shaking for three minutes in a shaking machine, while a mortar should be used for wet particulate samples. Other solid samples like concrete may, if necessary, be crushed into small pieces with a mortar. Since chemical warfare agents do not penetrate far into the material, the outer part of the sample is of most importance. Clothing, leather and polymers should be divided into sub-samples with a knife or scissors, and liquid samples well mixed before splitting.

- The extraction of the samples should be carried out in accordance with the recommended operating procedures for each sample material.

- The C_{18} cartridges brought back from the field should be eluted with 500μl of an organic solvent suitable for the final analysis. Acetone and dichloromethane have satisfactory eluting properties for chemical warfare agents and may be used for analysis by gas chromatography or mass spectrometry. For NMR or liquid chromatography, methanol may be used.

- The phosphonic acids are eluted from the NH_2 cartridges with 300 μl methanol. The eluates may be analyzed directly by HPLC or by GC after derivatization of the acids.

- The controls should be processed and analyzed in the same way as the samples in order to be sure that there has been no cross-contamination. The controls should also be spiked with any chemical warfare agent found to establish the recovery rate for the analytical method used.

- The recommended operating procedures for quality control for each instrument should be followed.

3 CHEMICAL PRODUCTION FACILITIES

3.1 Verification of location

Upon arrival at a chemical facility which is to be inspected, the position (coordinates) should be checked against the information given by the requesting state party to ensure that the correct area is examined. This should be done by comparing the terrain with a map, or by using navigational aids such as the global positioning system (GPS).

3.2 Where to take samples

The samples should be collected at the points specified in any facility agreement, or in the case of challenge inspection, at sites recommended by a process engineer in the inspection team. Samples most likely to contain information of interest are from feed stocks, process streams, reactors, storage tanks and waste. Samples from equipment not currently in use, but suspected to have been used for activities prohibited by the convention, may be collected by wiping with Whatman 41 filter paper.

3.3 Sampling

3.3.1 Recommended sample materials

The sample materials which are most likely to contain information of interest are:

- feed chemicals
- process liquids
- reaction mixtures
- products
- volatile and non-volatile waste (drainage ditches)
- wipe samples from equipment suspected to be contaminated with scheduled chemicals
- filter canisters used by personnel exposed to, or believed to be exposed to scheduled chemicals
- gasket materials

3.3.2 Numbers and sizes of samples

At least two samples should be collected from each sampling site in separate containers. The amount needed depends on the type of sample. For samples of the process steams and feed chemicals storage areas only a few grams are needed, but larger amounts (about 50 g) are needed for samples of waste. If the samples need to be split before analysis, larger samples should be collected to get the above-mentioned sizes of each sub-sample.

Controls should be collected to ensure that chemicals normally present in industrial samples do not interfere with the analysis of chemical warfare agents.

3.3.3 Sample containers

The most satisfactory sample containers are glass bottles with leakproof caps, but mylar bags or metallized plastic bags may also be used. The containers should fulfil the following requirements:

- The containers should not release any chemicals which cause contamination of the samples.

- It should not be possible for volatile compounds to escape from the containers.

- The containers should not consist of materials which strongly adsorb chemical warfare agents

- The containers should not accelerate the breakdown rate of chemical warfare agents.

- Gas samples should be passed through a column containing an adsorbent (e.g. Tenax) and should be placed in gas-tight containers.

3.3.4 Sampling procedure

To ensure the integrity of the samples, they should be collected by personnel from the chemical facility supervised by the inspection team. A spoon, spatula, scissors, knife, scoop and pipette should be available for sample collection. In addition, personnel carrying out the sampling should wear appropriate protective equipment. After sampling, the sample containers and sampling equipment should, if necessary, be decontaminated by washing with 5 % sodium hydroxide in a mixture of 2-propanol and water (1:1).

3.4 On-site analysis

The samples collected at the facility should, if possible, be analyzed on-site using in-house equipment or instrumentation brought by the inspection team. Simple equipment and methods such as CAM, detection paper and thin layer chromatography (see Chapter 2.4) may be used to screen for known chemical warfare agents. Electrical equipment has to be approved for use in the production facility.

3.5 Sample handling

3.5.1 Sample sealing

The samples should be sealed and secured immediately after collection to prevent loss or tampering before analysis. The plastic bags and glass bottles used for sampling should be sealed with a lead seal to prevent tampering during transport and storage. It is also important that the containers used for sampling are air tight to prevent any loss of sample.

3.5.2 Sample coding and documentation

Each sample should receive a unique identification code, and all information should be recorded in a sample documentation form with corresponding coding. The first two pages of the three-page form described in Chapter 2.5.2 may be used and completed with the following information:

- On the first page, information on the sampling procedure is entered, including the reason for sampling, an indication of the priority or importance of the samples, description of samples and sampling site, sampling methods and results of on-site analyses.

- On the second page, a sketch of the relevant part of the chemical production facility should be drawn, indicating pipelines, reactors and the positions of the sampling sites.

Three copies of the form should be made, one of which should accompany the samples to the Technical Secretariat, one should be left with the state party inspected and one should be kept by the sampling team.

3.5.3 Sample preservation

All samples should be treated as soon as possible after collection, to ensure that the chemical warfare agents are still present on arrival at the laboratory. The appropriate treatment varies according to the origin of the samples. To ensure the integrity of the samples, in some situation, no treatment is desirable.

- Aqueous samples (e.g. waste water) should be preserved by passing the sample through a cartridge filled with a polymer C_{18} adsorbent which retains most of the scheduled chemicals. The cartridge may then be transported to the laboratory for subsequent elution and analysis.

Procedure:

Pass 50 ml of the aqueous sample through a 200 mg C_{18} cartridge which has been pre-wetted with 0.5 ml methanol and 5 ml water. In order to prevent clogging of the cartridge, samples containing large amounts of particulate matter should be filtered through a 20 μm pore-size frit filter or through a Whatman microfibre filter grade GF/A.

- Phosphonic acids, which are decomposition products from nerve agents, are preserved by sorption to an aminopropyl weak anion exchanger (NH_2). Cartridges filled with 100 mg NH_2 material (Analytichem International Inc.) are attached after the C_{18} cartridges and the sample solution is passed through the combined cartridges. The chemical warfare agents are retained on the C_{18} cartridge (top) and the phosphonic acids on the NH_2 cartridge (bottom).

3.6 Sample transport

All samples should be properly secured to avoid injury to personnel handling the samples. This means that all samples should be transported surrounded by activated charcoal in a solid shock-resistant container. In addition, the samples should be treated in accordance with the following guidelines:

- To prevent degradation of chemical warfare agents during transport and storage, both the untreated samples and the C_{18} cartridges should be kept cold, preferably in a box filled with dry ice (-78.5°C). A freezing mixture, for example sodium chloride:ice = 1:3 (-21.3°C) or calcium chloride:ice (min. -55°C) could also be used.

- Liquid samples should not be frozen but should be transported cold in an insulated box with cooling elements.

- Each container should be properly packed and labelled according to the "Technical Instructions for the Safe Transport of Dangerous Goods by Air" (ICAO Doc 9284-AN/905). The shipping names: poisonous solid n.o.s or poisonous liquid n.o.s in class 6.1 (UN no. 2811 and UN no. 2810 respectively), may be used for samples containing only traces of chemical warfare agents. These compounds can be transported by passenger aircraft, except for those with an inhalation toxicity of Packing Group I (Great danger). It should be noted, however, that only boxes which are type approved for transport of dangerous goods may be used. A completed copy of the Shipper's Declaration for Dangerous Goods is shown in annex 2 to this report.

- A transport log should follow each parcel and be filled in by the personnel in charge of each part of the transport from the sampling site to the destination in order to maintain an unbroken chain of custody. The maximum permissible temperature during transport should be filled in by the sampling team as information for the personnel in charge of transport. A transport log has been drawn up and is shown as annex 3 to this report.

3.7 Sample handling in the laboratory

When the samples arrive at the laboratory, they should be treated in such a way that all information contained in the samples remains intact until the final analysis. This means that the following instructions should be followed:

- Upon arrival at the laboratory, the samples should be stored in a safe and tamper-proof place. Solid samples should be stored at -20°C or -70°C in a freezer and liquid samples preferably at +4°C in a refrigerator. The analysis should be carried out as soon as possible. If re-coding is necessary, all information given on the sample documentation form and in the transport log should be linked with the new coding.

- Before the main sample is split into sub-samples, it should be properly homogenized. Dry particulate samples should be homogenized by shaking for three minutes in a shaking machine, while a mortar should be used for wet particulate samples. Other solid samples may be crushed into small pieces with a mortar, and liquid samples well mixed before splitting.

- The extraction of the samples should be carried out in accordance with the recommended operating procedures for each sample material.

- The C_{18} cartridges brought back from the facility should be eluted with 500 μl of an organic solvent which is suitable for the final analysis. Acetone and dichloromethane have satisfactory eluting properties for chemical warfare agents and may be used for analysis by gas chromatography or mass spectrometry. For NMR or liquid chromatography methanol may be used.

- The phosphonic acids are eluted from the NH_2 cartridges with 300 μl methanol. The eluates may be analyzed directly by HPLC or by GC after derivatization of the acids.

- The controls should be processed and analyzed in the same way as the samples in order to be sure that there has been no cross-contamination. The controls should also be spiked with any chemical warfare agent found to establish the recovery rate for the analytical method used.

- The recommended operating procedures for quality control for each instrument should be followed.

4 LIST OF EQUIPMENT

The equipment needed for sampling, field analysis and sample handling in the field before transport should be packed in suitable (aluminum) boxes for easy transport to the sampling area, each box containing the equipment needed to carry out one part of the sampling and sample preparation procedure. If any part of the procedure is to be omitted, the corresponding equipment may be left behind without repacking.

The suggested composition of the boxes is listed below.

Box A: Equipment for sample collection

24 glass bottles (250ml) with screw caps
25 mylar bags
25 labels
1 box Whatman 41 filter paper
1 measuring cylinder (100 ml)
2 spoons
1 spatula
1 pair of scissors
1 scalpel
2 funnels
1 spray candle paint to mark the contaminated area
1 thermometer
1 measuring tape
equipment for sealing the samples
stationery
adhesive tape
10 sample documentation forms
5 transport logs

Box B: Equipment for solid-phase extraction

30 C_{18} cartridges (200 mg sorbent)
30 NH_2 cartridges (100 mg sorbent)
4 adapters
2 vacuum equipment (model NDRE)
1 microlitre pipette with tips (5 ml)
1 vacuum pump (12 VDC) with cable
1 thermos
8 glass bottles (50 ml) with screw caps
1 pair of tweezers
1 box Whatman filter GF-A
250 ml methanol
1 measuring cylinder (100 ml)
stationery

Box C: Equipment for TLC

250 ml chloroform
2 separating chambers with teflon covers
10 blotting papers
15 TLC plates
microcapillary applicators
1 spray equipment
1 microlitre pipette with tips (5 ml)
1 pair of tweezers
stationery
mobile phases
developing reagents

Box D: Protective equipment

Individual items: respirator
protective suit
2 pairs of boots
5 pairs of gloves
atropine/oxime autoinjectors
fullers' earth

1 box latex gloves (100 pieces)

Box E: Additional equipment

1 spraygun
1 spare bottle for spraygun
decontamination liquid (2-propanol/water/NaOH)
chloramine T
1 water can
1 funnel
1 measuring cylinder
1 primus
4 l kerosene
100 ml methylated spirit
1 adhesive tape (2-sided)
1 adhesive tape (1-sided)
1 box kleenex tissue paper
atropine/oxime autoinjectors
pyrido prophylactic
fullers' earth
10 plastic bags (for litter)
stationery

In addition the following equipment should be available:

map of the area under verification
GPS receiver (global positioning system)
chemical agent monitor (CAM)
detection paper
water analysis kit

5 REFERENCES

(1) Norwegian Working Paper on Verification of a Chemical Weapons Convention - Sampling and analysis of chemical warfare agents under winter conditions. Document CD/311 of 11 August 1982.

Research Report on Verification of a Chemical Weapons Convention. Sampling and analysis of chemical warfare agents under winter conditions. Annex to Document CD/311 of 11 August 1982.

(2) Norwegian Working Paper on Verification of a Chemical Weapons Convention - Sampling and analysis of chemical warfare agents under winter conditions. Document CD/396 of 19 July 1983.

Research Report on Verification of a Chemical Weapons Convention. Sampling and analysis of chemical warfare agents under winter conditions. Part II. Annex to Document CD/396 of 19 July 1983.

(3) Norwegian Working Paper on Verification of a Chemical Weapons Convention - Sampling and analysis of chemical warfare agents under winter conditions. Document CD/508 of 15 June 1984.

Research Report on Verification of a Chemical Weapons Convention. Sampling and analysis of chemical warfare agents under winter conditions. Part III. Presented as Document CD/509 of 15 June 1984.

(4) Norwegian Working Paper on Verification of a Chemical Weapons Convention - Sampling and analysis of chemical warfare agents under winter conditions. Document CD/600 of 20 June 1985.

Norwegian Working Paper on Alleged Use of Chemical Warfare Agents under Winter Conditions. Document CD/601 of 20 June 1985.

Research Report on Verification of a Chemical Weapons Convention. Sampling and analysis of chemical warfare agents under winter conditions. Part IV. Presented as Document CD/598 of 20 June 1985.

(5) Norwegian Working Paper on Verification of a Chemical Weapons Convention - Procedures for verification of alleged use of chemical weapons. Document CD/703 of 16 June 1986.

Norwegian Working Paper on Verification of a Chemical Weapons Convention - Evaluation of methods for identification of arsenic containing chemical warfare agents. Document CD/704 of 16 June 1986.

Research Report on Verification of a Chemical Weapons Convention. Sample handling of chemical warfare agents. Part V. Presented as Document CD/702 of 16 June 1986.

(6) Norwegian Working Paper on Verification of Alleged Use of Chemical Weapons - Summary of research results 1986/87. Document CD/761 of 24 June 1987.

Norwegian Working Paper on General Procedures for Verification of Alleged Use of Chemical Weapons for Consideration in the Negotiations on the Chemical Weapons Convention. Document CD/762 of 24 June 1987.

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(7) Norwegian Working Paper on Verification of Alleged Use of Chemical Warfare Agents. Document CD/861 of 22 August 1988.

Research Report on Verification of a Chemical Weapons Convention. Development of procedures for verification of alleged use of chemical warfare agents. Part VII. Presented as Document CD/857 of 12 August 1988.

(8) Norwegian Working Paper on Verification of Alleged Use of Chemical Weapons: A new approach for verification procedures. Document CD/936 of 21 July 1989.

Research Report on Verification of a Chemical Weapons Convention. Headspace Gas Chromatography - A new technique in verification of alleged use of chemical warfare agents. Part VIII. Presented as Document CD/940 of 31 July 1989.

(9) Report on a national trial inspection of an industrial chemical facility. Document CD/CW/WP.285 of 10 April 1990.

Norwegian Working Paper on Verification of Alleged Use of Chemical Weapons: Use of sorbent extraction in verification of alleged use of chemical weapons. Document CD/1008 of 26 June 1990.

Research Report on Verification of a Chemical Weapons Convention. Use of sorbent extraction in verification of alleged use of chemical weapons. Part IX. Presented as Document CD/1019 of 23 July 1990.

(10) Norwegian Working Paper on Verification of Alleged Use of Chemical Warfare Agents: Application of procedures after a simulated chemical attack on an air base. Document CD/CW/WP.340 of 30 May 1991.

Research Report on Verification of a Chemical Weapons Convention. Application of procedures after a simulated chemical attack on an air base. Part X. Presented as Document CD/1084 of 14 June 1991.

ANNEX 1 SAMPLE DOCUMENTATION FORM

SAMPLE DOCUMENTATION. SAMPLING.	Page 1 of 3

Items 1-11 refer to the sampling procedure.

1. Name : John Tørnes Date : 31.10.90 Time : 09:20

   Position : Scientist

2. Reason for sampling :
   Suspected attack on airbase with chemical weapons

3. Sample priority : First ☒ Second ☐ Third ☐

4. Sample code number :	d 3110-4
a 3110-1	e
b 3110-2	f
c 3110-3	g
5. Description of sample :	d Water
a Soil	e
b Sand	f
c Protective clothing from leg	g
6. Method of obtaining the sample(s) :	d Surface water
a Spooned from the surface	e
b Spooned from the surface	f
c Scissors	g

7. Results of field analysis (CAM, TLC) :

   CAM positive response in G mode
   TLC positive response for GA, GB, L, VX and H

8. Weather conditions :
   Partly cloudy, good weather, no precipitation
   Wind 14 kts from SSE, temperature 8°C

9. Description of sample site (topography, vegetation etc) :

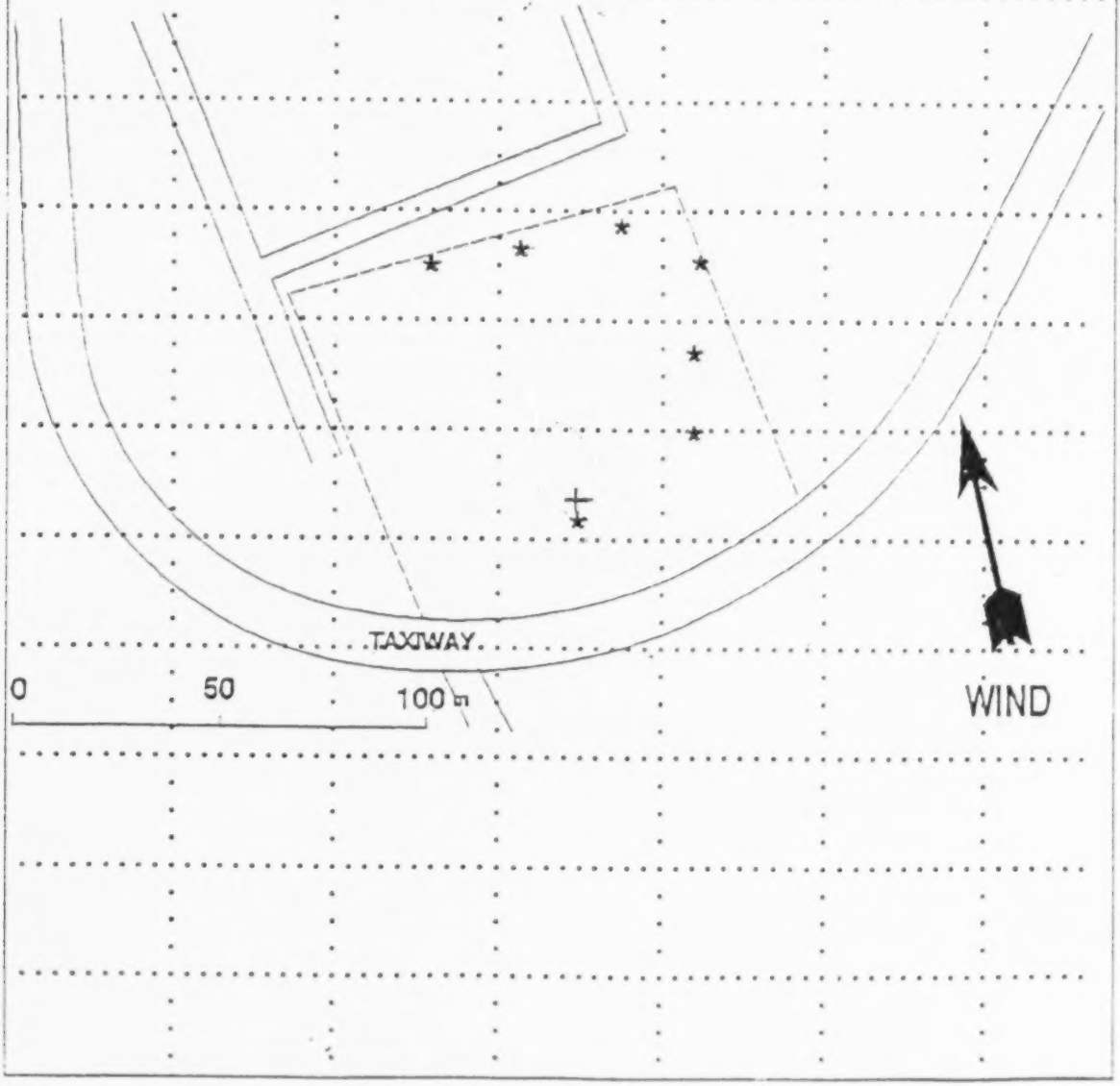
   Relatively flat field with black soil covered with long grass and low birches

10. Location of sample site (map reference UTM) :

    Northing: Easting:

SAMPLE DOCUMENTATION. SAMPLING. Page 2 of 3

11. Sketch of site , topography, bomb craters etc.
Use cross and sample code number to give sample site.
Please indicate North-South and wind direction.



SAMPLE DOCUMENTATION. ATTACK.	Page 3 of 3
Items 12-17 refer to observations concerning the attack.	
12. Date :	Time :
13. Weather conditions since the attack :	
14. Symptoms of casualties (humans/animals) :	
15. Effect on vegetation :	
16. Description of the attack method of delivery, smell, sound etc)	
17. Description of munitions found at the site :	
18. Remarks :	

ANNEX 2 SHIPPER'S DECLARATION

Shippers Declaration for Dangerous Goods

Shipper

NORWEGIAN DEFENCE RESEARCH
Establishment
P.O. Box 25,

N-2007 KJELLER, NORWAY

Air Waybill No.

Page of Pages

Shipper's Reference Number

Consignee

ANDØYA AIRPORT
Andenes

Norway

Two completed and signed copies of this Declaration must be handed to the operator

WARNING

Failure to comply in all respects with the applicable Dangerous Goods Regulations may be in breach of the applicable law, subject to legal penalities. This Declaration must not, in any circumstances, be completed and/or signed by a consolidator, a forwarder or an IATA cargo agent.

TRANSPORT DETAILS

This shipment is within the limitations prescribed for (delete non-applicable)

PASSENGER AND CARGO AIRCRAFT ~~CARGO AIRCRAFT ONLY~~

Airport of Departure: Oslo, Fornebu

Airport of Destination: ANDENES

Shipment type (delete non-applicable)

NON-RADIOACTIVE ~~RADIOACTIVE~~

NATURE AND QUANTITY OF DANGEROUS GOODS (see sub-Section 8.1 of IATA Dangerous Goods Regulations)

Dangerous Goods Identification						
Proper Shipping Name	Class or Division	UN or ID No.	Subsidiary risk	Quantity and type of packing	Packing inst.	Authorization
Poisonous solids n.o.s. (Environmental samples containing chemical warfare agents)	6.1	UN2811		1 Plywood box x 2 kg	606	
Poisonous liquids n.o.s. (Water containg chemical warfare agents)	6.1	UN2810		1 Plywood box x 1 l	601	

Additional Handling Information

I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked and labelled, and are in all respects in the proper condition for transport by air according to the applicable International and National Government Regulations.

Name/Title of Signatory

Place and Date

Signature (see warning above)

ANNEX 3 TRANSPORT LOG

TRANSPORT LOG.

1. Sampling carried out by : B.A.Johnsen

   Confirmed by : P.J.Karlsen

   Date : 31/10-90 Time : 09:20 Parcel number : 1

   Total number of parcels in shipment : 1

   Temp should not exceed : 10 °C Description of parcel : Wooden case

2. Transport and storage information.

Date and time received	Place received	Means of transport	Temperature during transport	Name and position
31/10-90 12:30	Airbase terminal	Car	8	Terminal leader
31/10-90 19:00	Oslo airport	Military plane	25	Private M.Plassen
31/10-90 19:30	NDRE Kjeller	Car	10	Scient. J.Tørnes

3. Received by J.Aa.Tørnes (sign)

   Institution NDRE Date : 31/10-90 Time : 19:30